

**Investigation on Composition,
Structure and Technology of Gypsum
Plaster for Contemporary
Reconstructure of Ancient Wall
Painting Renderings**

by

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Summary

This paper contains results of tests of gypsum plaster samples taken from Merenptah and Menena tombs from the 19th and 18th dynasties respectively, (New Kingdom). The comparison of their composition, structure and types of their components proves that the technology of their preparation was in both cases similar. The differences are only in quantitative and specific additive materials which improve plaster properties. It is fact that, local environment conditions and salt crystallisation which played an important role in the collapse of many wall painting renderings in the tombs, which exist in the valley of the kings and El-Hoza cemetery in Luxor. The aim of the present work is to reconstruct, fill gaps and fix the plaster with a suitable and appropriate new one.

Introduction

The application of gypsum plaster originated in ancient times in Egypt Gypsum mortar had also been used as a binder of the internal stone elements in the pharao's tombs, temples and the pyramid of Cheops, Chefren and Miceren (2900-2750 BC). The technology of gypsum as a plaster had spread from Egypt to Babylon and Asia Minor via Greece to Rome and then to the north of Europe.⁽¹⁾ The king tombs in the valley of the kings and the noble tombs in Qurna and El-Hoza Cemetery located on the west bank of the Nile in Luxor have been cut into poor quality hill rocks. The wall surfaces are rough and uneven. It was plastered with rendering of varying thickness over which a thinner plaster has been laid in order to remedy the irregularity and defect of the walls and to create smooth surfaces to receive the paint layer.

The painting was done in Merenptah and Menena Tomb on thinner - dry gypsum plaster. The pigments used are red, yellow ochre, Egyptian blue, white (gypsum and calcium carbonate) while the green colouring consists of calcium, silicon, copper and iron trace.

An initial indication of the main mineralogical composition of the historic plaster was obtained from the results of IR spectral, Derivative thermal analysis, X-ray diffraction, petrographic analysis of thin slices and cross sections technique.

It was found that historical plaster samples collected from Merenptah and Menena Tomb which have been examined are almost identical. In general, gypsum and sometimes small amounts of calcite were found to be a binder, while quartz, red ceramic grains (ceramic debris), carbonate and vegetable coal (aggregate) (Fot. 1) as additives gave plasters particular properties. Fibrous organic materials such as straw and husks were also identified by the naked eye and optical microscope (Fogt. 2).

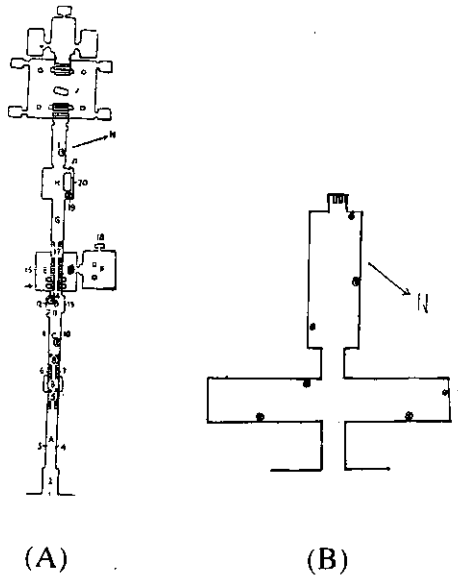
Fibrous organic materials, quartz, red ceramic powders and other fillers increased compressive strength and minimized cracking when the plaster dried.⁽²⁾

In general, the gypsum plaster in Merenptah and Menena Tomb has survived is still intact and is adhering well to the rock surface.

In spite of this general statement, plaster has fallen in several places from the walls due to the following reasons :

1. Effect of rains leaking through the crevices and cracks in the hill rock;
2. The effect of salts on and within the plaster layers;
3. There are detachments of the plaster in some areas resulting in bulges which can be seen in different places;
4. Local environments especially temperature fluctuation (diurnal and seasonal).⁽³⁾

Experimental works have been carried out to find suitable plaster with properties nearly similar to the original one in order to reconstruct, fill gaps and treat the misshaped and missing plasters.



Planes of Merenptah (A) and Menena Tombs (B) show the places from where the samples were taken.

Investigations :

Samples from Menena and Merenptah Tomb (New Kingdom 18th and 19th dynasties) were studied. (Plan A, B). In all areas where samples were taken from Merenptah Tomb, the plaster was composed of two layers, a coarser one of varying thickness and a finer smoother layer of 0.05 – 0.1 mm thick (Fig. 1). The samples had a reddish to

pink colour and their structure rather soft and weak. Samples taken from Menena Tomb composed also of two layers a coarser arriccio (with numerous vegetable straws and husks) varying in thickness topped with finer and smooth intonaco of 0.04 – 0.1 mm thick. The samples had pale-yellowish colour and are friable (Fig. 2).

Analytical methods :

The diagnosis of weathering phenomena and the choice of conservation methods require an understanding of the composition and structure of the original materials. For this, the following tests were carried out :

– X-ray Diffraction

Analysis carried out by using Russian apparatus “Dron-1” under the following conditions :

- Filterate radiation $CuK\alpha$
- Working parameters rtg lamp 20 mA°, 42 kv;
- Angular rate diffractometer arm : 1° /min.;
- Paper shift rate in registrator : 10 mm/min.

– Differential Thermal Analysis

Tests were carried out by using Hungarian apparatus “MOM” according to the following measurement conditions :

Samples	Temperature 20°C – 1000°C
– weight :	500 – 900 mg
– TG :	200 mg
– DTA :	250 μ v
– DTG :	500 μ v

– IR Spectral Analysis

Specord 75 IR apparatus produced in Germany was used. The pressed tablets prepared for analysis consisted of 250 mg of KBr and 2.5 mg of samples, or 40 mg of KBr and 0.4 mg of sample. Spectra of

— Investigation on Composition, Structure and Technology of Gypsum Plaster
the tablets were recorded in the wave number range 4000 Cm^{-1} to 400 Cm^{-1} .

- Thin Layer Chromatograph

No organic binder was detected in plaster samples.⁽⁴⁾

- **Petrographic Analysis** of thin slices, the tests of the mineralogical composition of thin sections in a passing light showed that the inorganic binder of plaster samples collected from Merenptah and Menena Tombs (historical plaster) is gypsum containing some fillers (a polarising microscope was used).

Results

Results of the tests on gypsum plasters from the Merenptah Tomb in the valley of the Kings in the west bank of the Nile, Luxor.

The samples show a micro-porous plaster, well compacted with grains of white alabaster and numerous red ceramic grains in the whole substance of the plaster. The mineralogical composition of plasters that have been examined is almost identical. In general, gypsum and sometimes small amounts of calcite act as (binder). The gypsum binder is fibrous in structure and has reddish-orange iron oxides (X-Ray diffraction shows gypsum and hematite) and carbonate incrustations. The existence of calcite in the plaster may intentionally have been added to the gypsum mortar or may have been connected with the presence of a small amount of calcium carbonate in the gypsum rock. Quartz occurs in the form of roundish grains, their size do not exceed 0.1 mms. Feldspar and very scarce, plates of mica exist (as aggregate). Plaster samples also contain roundish graing of slightly burnt clay, loamy minerals with dimensions ranging from 0.1 – 0.5 mm and brown roundish-longish grains of coal (vegetable coal). This indicates a low temperature of clay – kilning.⁽⁵⁾

Numerous red ceramics grains in the plaster were noticed. They do not contain any grains of quartz in their substance. In addition, a few fibrous organic materials (straw) from 1.2 to 2.2 mm are seen clearly und optical microscope though cross-sections. The average quantitative

analysis of plaster samples show 41.22% gypsum and 4.58% calcite. The rest are quartz, clay minerals and Remainders R_2O_3 51.07% (see table 1). The ratio between binder to fillers 1 : 1. Table (2) and pattern (1) illustrate the data given by IR spectrum. The chemical analysis proved that the amount of soluble salts in the historic plaster range from 5.0% – 7.5%.

Samples from	Humidity and/or Combined water %	Gypsum $CaSO_4 \cdot 2H_2O$ 110°C–190°C %	Calcite $CaCO_3$ 600°C–1000°C %	Remainder R_2O_3 %	Total
Merenptah tomb	from 20°C–110°C 1.34 and from 190°C–600°C 1.79	41.22	4.58	51.07	100%
Menena tomb	1.20	70.82	12.63	15.35	100%

Table 1 : D.T.A. data of gypsum plaster from Merenptah and Menena Tombs.

Plaster from Menena Tomb :

The tests of the mineralogical content of thin slices in a passing light showed that the plaster is gypsum containing some fillers. Gypsum binder is well crystallized. There is also crystalline calcite which form a kind of incrustations. Similar to plaster from Merenptah Tomb the existence of calcite in gypsum plaster may intentionally have been added or may have been connected with the presence of small amounts of calcium carbonate in the gypsum rock.⁽⁶⁾

The plaster contains few roundish grains of red ceramic containing ferrous substances and feldspar. Apart from gypsum, calcite and ceramic grains, quartz sharp edged grains whose dimensions do not exceed 0.4 mm, fine grains of alabaster, clay and brown coal grains were identified.

In addition, very long fibrous organic materials (straw or husks) from 5 mm to 8 mm can be recognised by the naked eye. Not similar to plaster taken from Merenptah Tomb, quantitative analysis of Menena Tomb shows higher content of gypsum – 70.82%, calcite 12.63% and quartz together with clay minerals R_2O_3 15.35% (Table 1). Table 2 illustrates the data obtained by IR analysis. The chemical analysis show the presence of chloride salts e.g. sodium chlorides.

Samples from	Peak Intensity Cm^{-1}	Mineral	Intensity
Merenptah Tomb	3550 - 3400 - 3225 - 1680 - 1625 - 1140 - 1120 - 670 - 600 1100 - 1000 - 800 - 780 1100 - 1040 - 915 1440 - 875 - 712	Gypsum Quartz Silicate or alumino- silicate Calcite	Big Small Small Very small
Menna Tomb	3550 - 3400 - 3250 - 2250 - 1695 - 1150 - 1130 - 670 - 600 2520 - 1795 - 1430 - 879 - 712 806, 480 3690 - 1030 - 940 - 910	Gypsum Calcite Quartz Clay min.	Big Small Small Very small

Table (2) : IR spectrum results of historical plaster from Menena and Merenptah Tombs.

Preparation of the proposed plaster recipes :

Based on both the analysis of old plasters of Merenptah and Menena Tombs and existing experience, the following materials and additives were used to prepare tentative plaster samples (Fot. 3) : kaoline $Al_2Si_2O_5(OH)_4$, chalk $CaCO_3$, sand SiO_2 (max. size 1 mm), Malta (6001 and 6002 consisted of : SiO_2 , CaO , AlO_2O_3 and pfuoco), red brick powder (max. size up to 1 mm), black charcoal and lime.

Malta and red brick powder were used in order to meet the strength requirements of the consolidation of the monuments and they increased compressive strength. At the same time ceramic powder and black charcoal contributes to strength and prevents capilarity, as well as, increasing the water retentivity of plaster (7). Such specific character is very important to the plaster and mortars existing in the arid dry climate (dry environments can cause exfoliation, micro and macro cracks to the new plaster). The composition and mex proportions of the plaster samples are given in Table (3).

Plaster No.	Kaoline.	Chalk	Sand max size 1 mm	Lime	Malta 6001	Malta 6002	Red ceramic Powder max size 1 mm	Black charcoal	Compressive st. MPa
1 p			3	1					0.90
2 p			3	1	0.75				1.90
3 p			0.50	1		1			0.80
4 p	1		1.50		0.25		0.20	0.03	2.54
5 p	1		0.60	1			0.10	0.03	0.19
6 p		1	0.60	0.60			0.10	0.03	0.15
7 p	1	1	0.60			0.30	0.15	0.03	0.40
8 p		1	0.60		0.75		0.08	0.03	
9 p	2	1	0.50		0.70		0.10	0.03	0.47
10p	1		2	0.50			0.25	0.03	0.70

Table (3) : Mix proportions of the plaster (mortar).

Conclusions

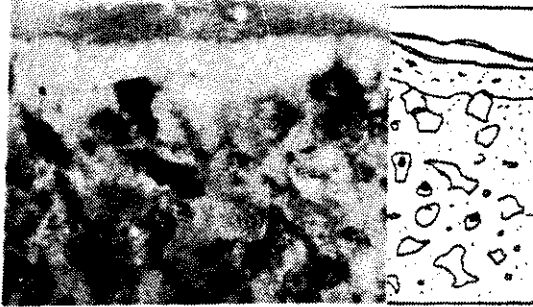
Gypsum plaster samples taken from Merenptah and Menena Tombs show a very high degree of similarity which manifests itself in their structures, fillers and technology. Differences are only in quantitative raw materials. Both tombs have been effected by soluble salts and arid environments.

In spite of the good and intact adherence of the wall painting to the rock surface of the tombs, the plaster has fallen very considerably from the walls in several places. The aim of the present work is to reconstruct and fill such defects with suitable new plaster. Of course the aim is not to copy historic plasters completely. Instead one should use the possibility of obtaining promising plaster with properties compatible with the historical plaster. The restorers and conservators in Egypt used

to add and mix polymers or synthetic emulsions to the new plaster or mortar, but in very dry climates such additives always lead to cracks and in serious conditions exfoliation and detachment of the reconstructing plaster (Fot. 4, 5). As a result, plaster samples of various proportions and recipes have been tested as shown in Table 3. It was found that samples 4P and 10P are the best for the reconstructure work, while 7P and 9P are good but they have lower compressive strength – 0.40 MPa and 0.47 MPa respectively. Samples 5P, 6P and 8P are fragile and do not resist even fracture by fingers.

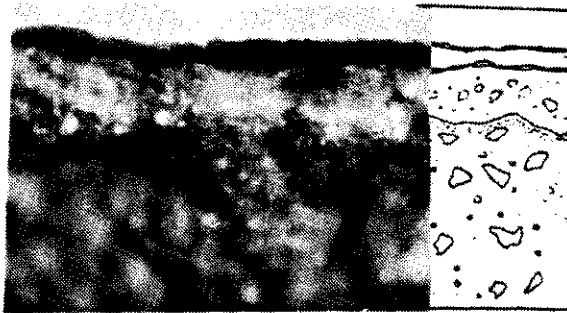
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- (C) paint layer
- (B) finer-smooth
gypsum layer
0.2-0.4mm
- (A) coarser
gypsum layer
5-7 mm.

Fig. (1)

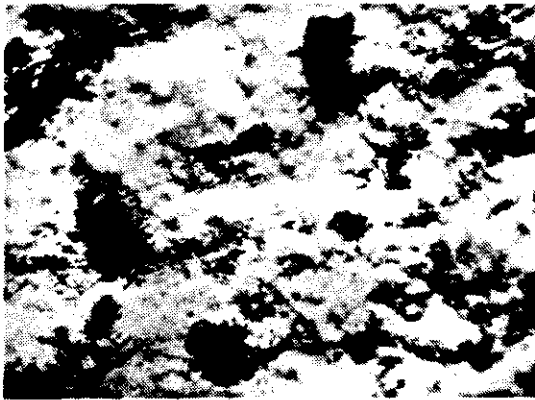


- (C) paint layer
- (B) finer-smooth
gypsum
prime layer
0.3 - 0.5 mm
- (A) coarser
gypsum layer
3-4 mm.

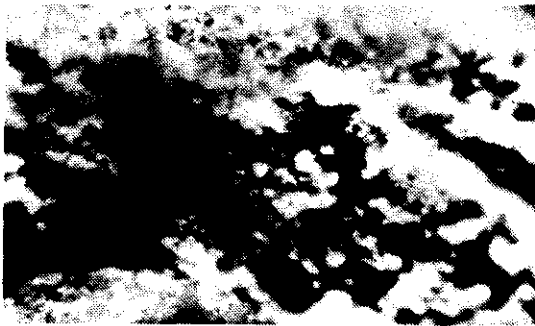
Fig. (2)

Photo and diagrams showing the main strata forming the wall painting.

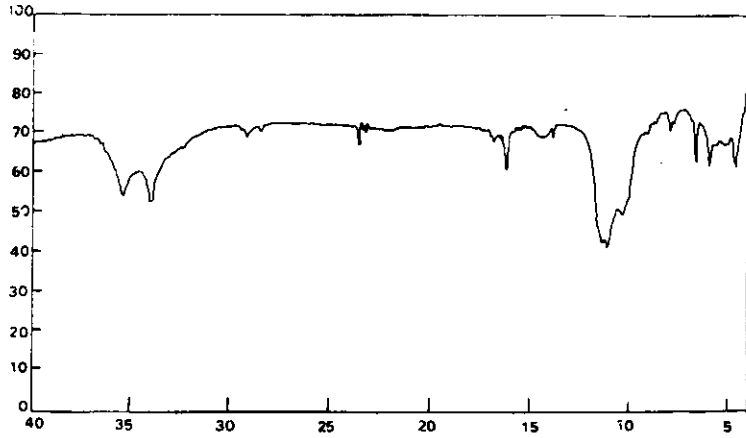
- (1) Cross-section from Merenptah Tomb
- (2) Cross-section from Menena Tomb.



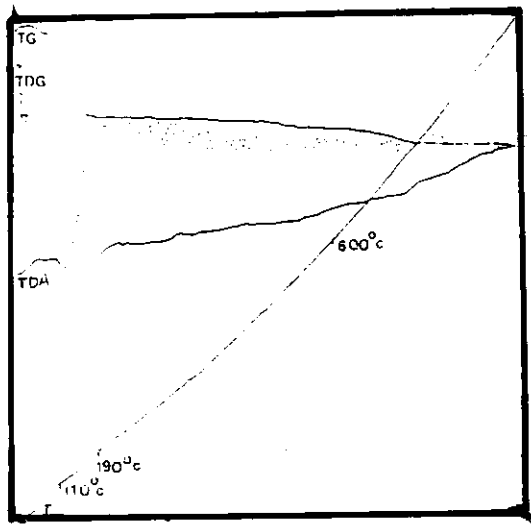
Fot. (1) : Red ceramic, brown coal and charcoal grains – under microscope (From the Merenptah Tomb).



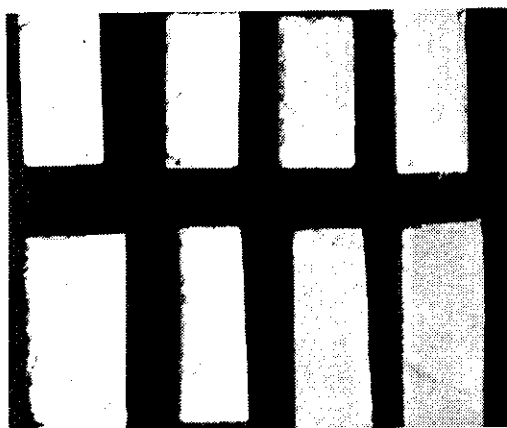
Fot. (2) : Fibrous organic materials (straw) within gypsum plaster.



Pattern (1): IR spectral of gypsum samples from Merenptah Tomb.



Pattern (2): Differential T.A. of gypsum plaster from Menena Tomb.



Fot. (3) : Tentative plaster samples.



Fot. (4), (5) : The old restoration (Menena Tomb).